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"STRENGTHENING OF CHROMIUM-MAGNESIA COMPOSITES"

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Department of the Navy, Bureau of Naval Weapons

Contract N600 (19) 59647 (C.P.F.F.)

Interim Report No. 2

Covering Period 1 April 1963 through 31 May 1963

BENDIX PRODUCTS AEROSPACE DIVISION
THE BENDIX CORPORATION
SOUTH BEND 20, INDIANA

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Prepared By: G. C. Reed

Approved By: W. L. Schalliol

ABSTRACT

This report describes the work accomplished during the second bi-monthly period of a program aimed at strengthening chromium-ceramic composites by alloying. During the previous reporting period, the notch tensile properties of Chrome-30 were evaluated at several stress concentration levels. Extrusions were prepared for evaluation of several compositional and processing variables aimed at improving ductility prior to initiation of the alloying study. The impact and tensile properties exhibited by these extrusions have now been determined and are reported herein. Pertinent chemical analyses are also reported. The best improvements were obtained by reducing the magnesia content. The compound containing three weight percent magnesia exhibited 45% tensile elongation at room temperature. It was decided that these results warranted further study before starting the alloy phase of this program. Four billets representing other low magnesia percentages have been prepared for this purpose.

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1.0 INTRODUCTION

The purpose of this program is to investigate alloy strengthening of chromium-ceramic composites while maintaining useable ductility. As pointed out in the first interim report, the approach selected called for the optimization of ductility before proceeding with the alloying investigation. This approach is dictated by the expectation that the alloying will result in some loss in ductility. The first interim report covered the determination of the notch tensile properties of Chrome-30, a chromium-magnesia-titanium composite representative of current production material. The properties of this material will furnish a basis for comparison with the compositional and processing variations evaluated throughout the rest of this program.

The work described in this report was accomplished during the period April 1, 1963-May 31, 1963. It consists primarily of the evaluation of seven different chromium-composite extrusions aimed at improving ductility.

2.0 EVALUATION OF EXTRUSIONS AIMED AT IMPROVING DUCTILITY

2.1 Composition and Process Variables Investigated:

The seven variables included in this study are:

2.1.1 Four variations in MgO content (0, 3, 6, & 9 weight %) aimed at minimizing the stress concentration effect of the oxide particles while retaining the beneficial aspects thereof.

2.1.2 Two processing variations aimed at reducing the oxide particle size and improving dispersion. The first of these variations was a reduction in sintering temperature so as to increase porosity. This was intended to allow greater relative particle motion during extrusion which, in turn, would break up the ceramic particles and disperse them in the chromium matrix. The other processing variation tried involved heating the MgO powder just prior to blending in an effort to drive off the chemical water and reduce the size of the agglomerates. It was felt that the purchase of an extremely fine MgO powder would be futile since it would agglomerate as soon as the container was opened due to the hygroscopic nature of the material.

2.1.3 One substitute for MgO, ThO_2, was tried to evaluate the effect of a more inert oxide dispersion in improving the resistance to oxide coalescence at elevated temperatures. The ThO_2 content was chosen to give the same volume percentage (5.86) as 3 weight % MgO.

2.1.4 In addition to these variations, it was also considered desirable to study the substitution of BeO for MgO. The BeO would offer resistance to coalescence and is also a spinel-former like MgO. It has been proposed that the formation of the spinel $MgCr_2O_4$ contributes to improved oxidation resistance and to matrix purification by acting as an impurity sink. Difficulties were encountered initially in finding an extrusion facility equipped to handle the billet containing the toxic BeO. Subsequently, Nuclear Metals, Inc., extruded it but the properties could not be evaluated in time for inclusion in this report. This extrusion contains 2.5 weight % BeO which is the same volume % as 3 weight % MgO.

2.2 Extrusion Procedures:

2.2.1 The seven billets included in this study were extruded at the ASD Metals and Ceramics Laboratory at Wright-Patterson AFB. All extrusions were successful. The billets were extruded at 2200°F using a ratio of approximately 10:1. Corning 0010 borosilicate glass was used for lubrication. The extrusions were in the form of flat bars roughly 0.4" thick by 1.8" wide.

2.3 Chemical Analyses:

2.3.1 Samples of each of the seven extrusions were sent to Ledoux & Company of Teaneck, New Jersey, for chemical analysis. A sample of the as-sintered billet consisting solely of chromium was also analyzed. The reported results are summarized and appended to this report (Section 5.1). It will be noted that the impurity levels are lower in the extrusions containing MgO than in those containing ThO_2 or no oxide addition. The explanation for this isn't clear since the analyses are reportedly "total contents" which would exclude the possibility of the MgO or $MgCr_2O_4$ acting as an impurity sink. Possibly the MgO promotes the removal of impurities during the sintering cycle. The analyses indicate that N and C are picked up during the handling operations prior to sintering. The excess carbon is then apparently removed during intering while the N content is uneffected.

2.4 Microstructures:

2.4.1 Microsections were prepared for each sintered billet and each extrusion. They revealed good dispersion of the ceramic particles in every case. The billets that had been sintered at a lower temperature exhibited a definitely smaller, well-dispersed ceramic phase. The hot MgO admix did not appear to have contributed any improvement beyond that due to reduced sintering temperature alone. The extrusion made from chromium without any additives exhibited a small amount of uniformly distributed chromium oxide throughout its internal structure. The microsection of the extrusion containing

ThO_2 revealed an evenly distributed mixture of chromium oxide and the somewhat larger and more angular ThO_2 particles.

2.5 Sintered and Extruded Densities:

2.5.1 The densities of each of the sintered billets and of the extrusions are tabulated in the Appendix (Section 5.2). The as-sintered billet densities were determined by weighing each machined billet and calculating its volume from dimensional measurements. The extrusion densities were determined for small representative specimens using the Archimedes method. It will be noted that all of the extruded densities were above 96% of the theoretical values.

2.6 Impact Testing:

2.6.1 Unnotched Izod bars 3" long x .394" x .315" were used for all of the impact testing in this investigation. A series of tests were first run with production Chrome-30 to provide a basis of comparison for the variables under study. The impact transition temperature was determined for Chrome-30 bars with as-ground surfaces (avg. = rms 30) and with polished surfaces (avg. = rms 3). The polished bars exhibited a transition temperature of 325°F; approximately 100°F below that of the bars with ground surfaces. Polishing these bars was very time consuming so it was decided to evaluate all of the extrusions under study with ground surfaces.

2.6.2 Six bars were prepared for each of the seven extrusions being investigated. They were dye checked for defects before testing. A 30 ft-lb hammer was used for these tests. The specimens were heated with a small resistance furnace while clamped in the vise of the impact tester. The specimen temperature was monitored with a thermocouple adjacent to it that had been carefully calibrated with a special test bar containing several thermocouples. This procedure assured an impact temperature measurement within ± 10°F. The Izod bars representing the seven extrusions were tested at several temperatures in an attempt to bracket the transition temperature. This was accomplished in all cases except for the compositions containing 9% MgO and 8% ThO_2 respectively. The data was inconclusive in the first case and no points above the transition were obtained in the latter case. The data for the entire series of impact tests is presented graphically in the Appendix (Section 5.3). It is noteworthy that both the 3% MgO and the 6% MgO compounds exhibited lower impact transition temperatures than the Chrome-30. One or more tests were rejected for each compound because of questionable dye check indications or improper test performance.

2.6.3 The impact tests were felt to be somewhat insensitive to the variations under study in that they did not clearly indicate which

of the variations was best. Therefore, it was decided to extend the investigation of these variations to include tensile tests.

2.7 Tensile Testing:

2.7.1 Three unnotched tensile specimens were prepared from each extrusion. A modified ASTM bar with a 3/4" gage length and a 0.189" diameter reduced section was used. The reduced section was polished by hand with emery paper to a finish of less than 20 rms. Electropolishing was not employed. A snap-on extensometer was used to record elongation during the test. The MAB standard procedure for refractory materials, #176-M, was followed. This calls for a strain rate of 0.005 in/in/min in the elastic region and 0.05 in/in/min in the plastic region. All tests were performed at room temperature.

2.7.2 The tensile test results are summarized in the Appendix (Section 5.4). A number of unusual findings were obtained. The Cr + 3% MgO compound exhibited up to 45% elongation at room temperature. This is almost a twofold increase in the ductility of Chrome-30. In addition, the yield and ultimate strength of Chrome-30 were retained. The reduced portion of these high-elongation specimens had an oval cross-section after testing. The Cr + 8% ThO_2 material exhibited no plastic deformation and only moderate ultimate strength. In most cases, a rather wide spread between the upper and lower yield was noted. The finer dispersion of oxide in the low-sintered Cr + 6% MgO extrusion gave a higher elongation value than Chrome-30 but the improvement wasn't as great as with the Cr + 3% MgO material. The latter material was also superior to the other MgO percentage variations. Pure chromium was almost as bad as the Cr + 8% ThO_2.

2.7.3 In view of the outstanding ductility exhibited by Cr + 3% MgO, it was agreed that further study of low oxide contents was warranted at this time. The alloying phase of the program will be temporarily postponed while MgO contents just above and below 3% are evaluated. This should permit the optimum MgO content from the standpoint of ductility to be established for incorporation in the alloy investigation. Four extrusions containing 1%, 2%, 4%, and 5% MgO are currently being processed. The properties of the Cr + 2.5% BeO extrusion will be evaluated along with these materials.

2.7.4 All of the tensile specimens described above were annealed for 1 hr. @1800°F in vacuum before testing. This heat treatment is intended solely for stress-relieving since the composites are recrystallized in the as-extruded condition. Since many potential applications would involve higher service temperatures, it was decided to evaluate the effects of higher annealing temperatures. A number of tensile specimens of both Chrome-30 and the Cr + 3% MgO compound are being prepared for this purpose.

3.0 CONTINUATION OF NOTCH TENSILE INVESTIGATION

3.1 Notch Tensile Properties of Unannealed Chrome-30:

3.1.1 The first interim report under this contract described the determination of the notch tensile properties of Chrome-30. The effects of various stress concentration levels and strain rates on the notched/unnotched ultimate strength were determined. The effect of annealing or not annealing after machining the notches was also investigated at a K_T of 3. A 20% decrease in the strength ratio was indicated for the unannealed test specimens. No studies of unannealed bars with notches having K_T's over 3 were tried since a post-machining, stress-relieving heat treatment is standard for most potential applications. It was subsequently decided that such tests would be of interest, however, in broadening the knowledge of this material. Consequently, a number of unannealed bars with notches in the K_T 5-8 range have been machined and tested. The loss in properties did not appear to be as great as was indicated at a K_T of 3. In several tests, the strengths were the same as for annealed bars. The data from these tests are plotted, along with the previous data, in Section 5.5 in the Appendix. A few errors found in the earlier data have been corrected in this report.

4.0 FUTURE PLANNING

4.1 The Following Work is Planned for the Next Reporting Period:

4.1.1 Evaluation of impact transition temperature of the 1%, 2%, 4%, and 5% MgO extrusions and the Cr + 2.5% BeO extrusion.

4.1.2 Evaluation of room temperature unnotched tensile properties of these five extrusions.

4.1.3 Determination of impurity contents of representative samples of these five extrusions.

4.1.4 Evaluation of notch tensile properties and oxidation resistance of the extrusion exhibiting the best combination of unnotched tensile ductility and strength.

4.1.5 Evaluation of the effects on tensile properties of stress-relieving heat treatments at temperatures up to 2500°F.

4.1.6 Selection of alloying parameters for solid solution strengthening investigation.

4.1.7 Preparation of extrusion billets required for solid solution strengthening investigation.

5.0 APPENDIX

(These items are found on the following pages)

5.1 Chemical Analyses of Materials Used in This Investigation.

5.2 Table of Densities of As-Sintered and As-Extruded Composites.

5.3 Impact Transition Temperatures of Extruded Composites.

5.4 Unnotched Tensile Properties of Extruded Composites.

5.5 Plot of Notched/Unnotched Ratio vs. K_T for Chrome-30.

5.6 Distribution List.

Compound Code Number	Nominal Composition (Weight %)	Elements Reported N (ppm)	O (%)	C (ppm)	S (ppm)	Mg (%)	Th (%)
As-Received Cr Powder		52	0.67	202	150	-	-
191.543	100% Cr (As-Sintered)	131	0.70	524	170	-	-
191.543EA107	100% Cr (Extruded)	133	0.63	180	240	-	-
192.567EA111	Cr+3% MgO (Extruded)	51	-	81	190	1.76	-
193.568EA112	Cr+6% MgO (Extruded)	60	-	95	190	3.18	-
194.547EA108	Cr+9% MgO (Extruded)	77	-	93	190	5.02	-
195.564EA109	Cr+8% ThO_2 (Extruded)	99	-	122	180	-	5.95
197.569EA113	Cr+6% MgO(H,R)* (Extruded)	91	-	102	180	3.31	-
198.542EA106	Cr+6% MgO(R)* (Extruded)	117	-	83	200	3.25	-

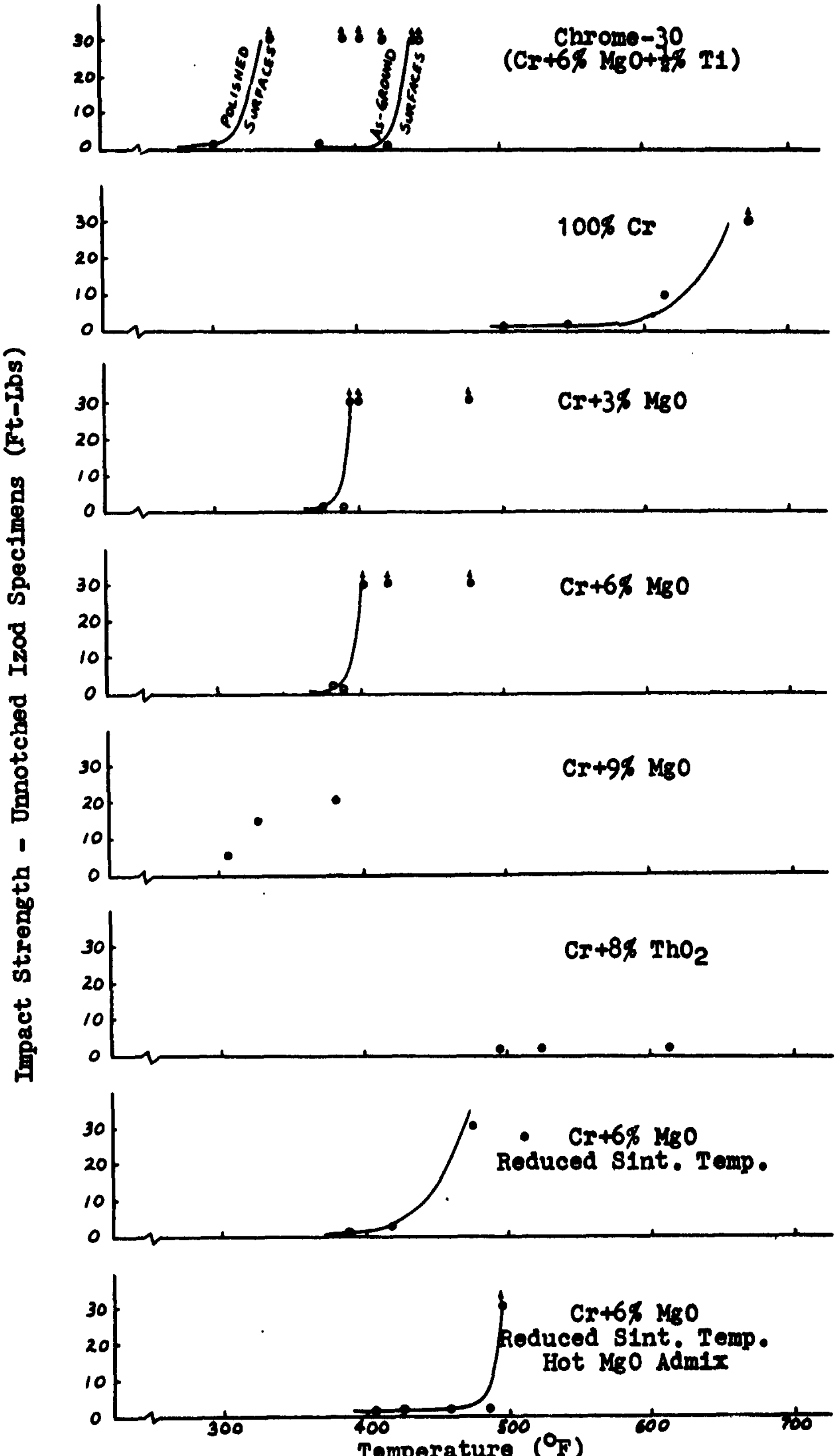
* "H" = hot MgO admix; "R" = reduced sintering temperature

5.1 Chemical Analyses of Materials Used in This Investigation

Compound Code	Nominal Weight % Composition	Sintered Density		Extruded Density	
		gm/cc	% Theoretical	gm/cc	% Theoretical
2400-191	100% Cr	5.27	74.4%	7.15	99.5%
2400-192	Cr+3% MgO	5.44	78.0%	6.87	98.6%
2400-193	Cr+6% MgO	5.46	80.6%	6.60	97.4%
2400-194	Cr+9% MgO	5.43	82.3%	6.35	96.2%
2400-195	Cr+8% ThO_2	5.82	79.0%	7.24	98.4%
2400-197	Cr+6% MgO(H, R)*	4.97	73.4%	6.56	96.8%
2400-198	Cr+6% MgO(R)*	5.70	84.1%	6.57	96.9%

* "H" = hot MgO admix; "R" = reduced sintering temperature

5.2 **Table of Densities of As-Sintered and As-Extruded Composites**

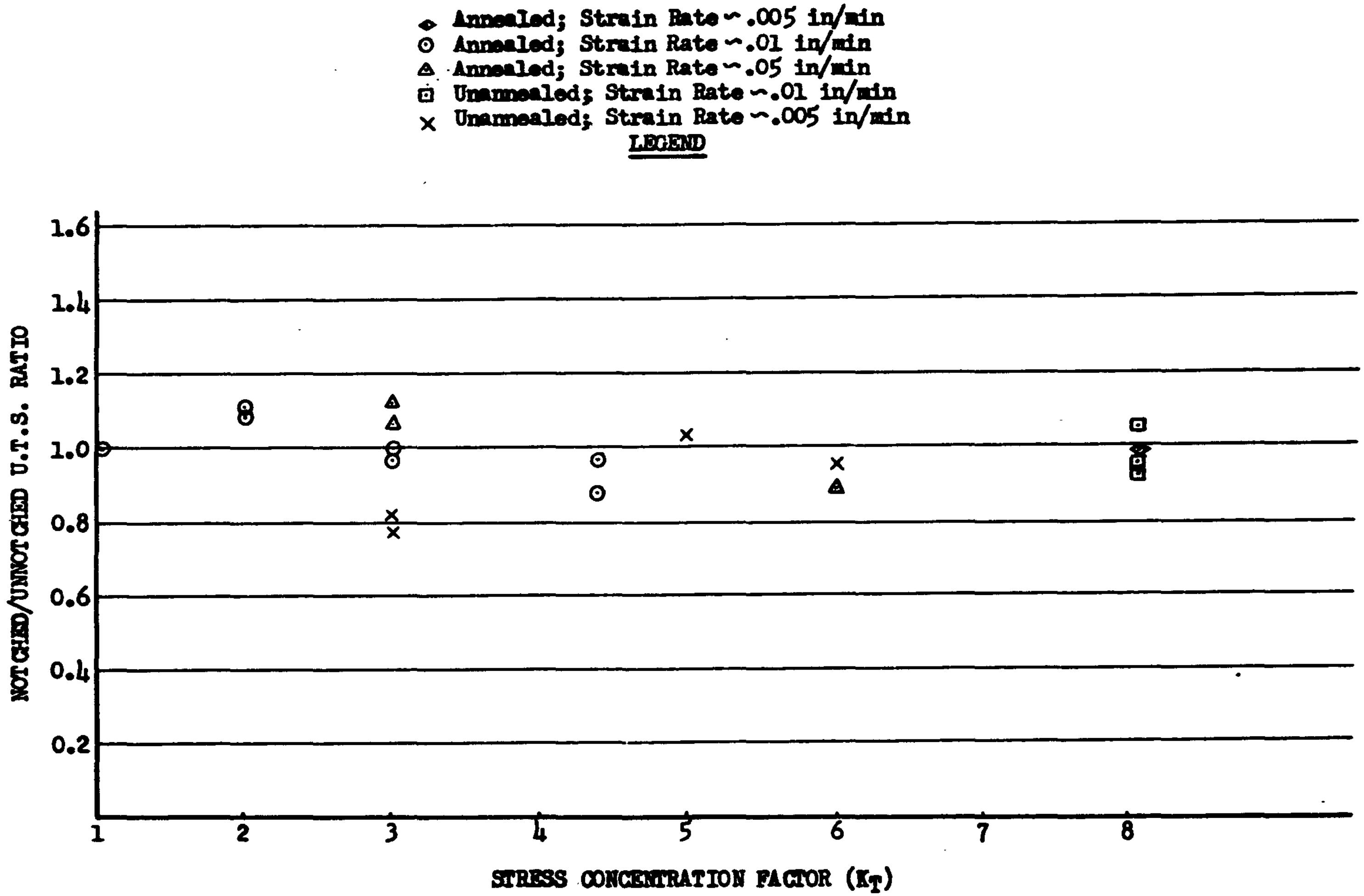


5.3 Impact Transition Temperatures of Extruded Composites

Test No.	Specimen Description	Yield (1000 PSI)	Ultimate (1000 PSI)	Elongation (% of G.L.)	Reduction Area (%)	Remarks
63-204	Cr	None	45.0	1.5	0	
63-205	Cr	44.5/24.0	44.5	6.4	6.0	Shoulder Break
63-206	Cr	None	48.5	2.0	0	
63-207	Cr	42.0/27.0	42.0	10.1	8.5	Shoulder Break
63-217	Cr+3% MgO	37.2/25.3	51.3	44.0	32.0	
63-218	Cr+3% MgO	30.6/21.6	49.0	45.0	32.0	
63-219	Cr+3% MgO	30.6/22.6	48.5	35.7	28.0	
63-220	Cr+6% MgO	28.4/22.0	45.0	26.3	16.0	
63-221	Cr+6% MgO	28.4/21.9	46.0	26.3	15.3	
63-222	Cr+6% MgO	28.4/21.9	45.5	26.3	15.3	
63-201	Cr+6% MgO(R)*	34.3/23.6	48.5	28.3	18.2	
63-202	Cr+6% MgO(R)*	31.0/24.5	48.2	17.7	9.4	Profilometer Indicates Rough Surface
63-203	Cr+6% MgO(R)*	36.0/24.0	48.4	26.4	16.3	
63-223	Cr+6% MgO(R,H)*	35.6/30.0	48.0	29.6	18.3	
63-224	Cr+6% MgO(R,H)*	40.0/26.0	47.5	30.6	17.5	
63-225	Cr+6% MgO(R,H)*	40.6/28.2	46.5	30.6	19.3	
63-208	Cr+9% MgO	22.0	41.8	16.4	8.2	
63-209	Cr+9% MgO	22.8	41.5	16.4	7.2	
63-210	Cr+9% MgO	22.5	41.5	18.1	8.2	
63-211	Cr+8% ThO_2	None	42.5	0	0	Shoulder Break
63-212	Cr+8% ThO_2	None	41.4	0	0	Shoulder Break
63-213	Cr+8% ThO_2	None	37.0	0	0	

* "H" = hot MgO admix; "R" = reduced sintering temperature

5.4 Unnotched Tensile Properties of Extruded Composites



5.5 Plot of Notched/Unnotched Ratio vs. K_T for Chrome-30
(Room Temperature Data)

5.6 <u>Distribution List - Contract N600(19)59647</u>:

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